

APPENDIX J

COMPARISON OF TARGET ANALYTE SCREENING VALUES (SVs) WITH DETECTION AND QUANTITATION LIMITS OF CURRENT ANALYTICAL METHODS

Table J-1. Comparison of Target Analyte Screening Values (SVs) with Reported Detection and Quantitation Limits of Current Analytical Methods^a

Target Analyte	SV ^b	Methods									
		Puget Sound Protocols ^c		National Study of Chemical Residues in Fish ^d		EMSL ^e	National Contaminant Biomonitoring Program ^f		California OEHHA ^g	State of California, Dept. of Fish and Game Environmental Services Division ^h	EPA 301(h) Monitoring Program ⁱ
		LOD ^j	PQL ^k	MLD ^l	TQL ^m	MDL ⁿ	LOD ^o	LOQ ^p	MDL ^q	LOD ^r	Detection Limits ^s
Metals											
Arsenic (inorganic) ^t	3 ppm	N/I	N/I	N/I	N/I	N/I	N/I	N/I	N/I	N/I	N/I
Cadmium	10 ppm	0.01 ppm	N/R	N/I	N/I	0.02 ppm	0.005-0.046 ppm	N/R	N/I	0.01-0.1 ppm	0.01 ppm (GFAA); 0.4 ppm (ICP)
Mercury	0.6 ppm	0.01 ppm	N/R	1.3 ppb (LOD) ^u	N/R	0.1 ppm	0.01-0.05 ppm	N/R	0.050 ppm	0.02 ppm	0.01 ppm (CVAA)
Selenium	50 ppm	N/I	N/I	N/I	N/I	0.6 ppm	0.017-0.15 ppm	N/R	N/I	0.05 ppm	0.02 ppm (GFAA)
Tributyltin	0.3 ppm	N/I	N/I	N/I	N/I	N/I	N/I	N/I	2.5 ppb ^v	0.05 ppm (dry weight) ^v	N/I
Organochlorine Pesticides						N/I					0.1-5 ppb ^w
Chlordane (total)	80 ppb										
cis-Chlordane		1-5 ppb	20 ppb	N/R	2.5 ppb		<1.5 ppb	2-15 ppb	3-5 ppb	5 ppb	
trans-Chlordane		N/I	N/I	N/R	2.5 ppb		<1.5 ppb	2-15 ppb	2-5 ppb	5 ppb	
cis-Nonachlor		N/I	N/I	N/R	2.5 ppb		<1.5 ppb	2-15 ppb	N/I	5 ppb	
trans-Nonachlor		N/I	N/I	N/R	2.5 ppb		<1.5 ppb	2-15 ppb	4-7 ppb	5 ppb	
Oxychlordane		N/I	N/I	N/R	2.5 ppb		<1.5 ppb	2-15 ppb	N/I	5 ppb	
DDT (total)	300 ppb								38 ppb		
4,4'-DDT		0.1-2 ppb	4 ppb	N/I	N/I		<1.5 ppb	2-15 ppb	7-13 ppb	10 ppb	
2,4'-DDT		0.1-2 ppb	4 ppb	N/I	N/I		<1.5 ppb	2-15 ppb	5-6 ppb	10 ppb	
4,4'-DDD		0.1-2 ppb	4 ppb	N/I	N/I		<1.5 ppb	2-15 ppb	5-6 ppb	10 ppb	
2,4'-DDD		0.1-2 ppb	4 ppb	N/I	N/I		<1.5 ppb	2-15 ppb	3-5 ppb	10 ppb	
4,4'-DDE		0.1-2 ppb	4 ppb	N/R	2.5 ppb		<1.5 ppb	2-15 ppb	15-38 ppb	5 ppb	
2,4'-DDE		0.1-2 ppb	4 ppb	N/I	N/I		<1.5 ppb	2-15 ppb	6-10 ppb	10 ppb	
Dicofol	10,000 ppb	N/I	N/I	N/R	2.5 ppb		N/I	N/I	N/I	100 ppb	
Dieldrin	7 ppb	0.1-2 ppb	4 ppb	N/R	2.5 ppb		<1.5 ppb	2-15 ppb	N/I	5 ppb	
Endosulfan (total)	60,000 ppb										
Endosulfan I		N/I	N/I	N/I	N/I		N/I	N/I	N/I	5 ppb	
Endosulfan II		N/I	N/I	N/I	N/I		N/I	N/I	N/I	70 ppb	
Endrin	3,000 ppb	N/I	N/I	N/R	2.5 ppb		<1-5 ppb	2-15 ppb	N/I	15 ppb	

See notes and references at end of table.

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Table J-1 (continued)

Target Analyte	SV ^b	Methods									
		Puget Sound Protocols ^c		National Study of Chemical Residues in Fish ^d		EMSL ^e	National Contaminant Biomonitoring Program ^f		California OEHHA ^g	State of California, Dept. of Fish and Game Environmental Services Division ^h	EPA 301(h) Monitoring Program ⁱ
		LOD ^j	PQL ^k	MLD ^l	TQL ^m	MDL ⁿ	LOD ^o	LOQ ^p	MDL ^q	LOD ^r	Detection Limits ^s
Organochlorine Pesticides (continued)											
Heptachlor epoxide	10 ppb	N/I	N/I	N/R	2.5 ppb		<1-5 ppb	2-15 ppb	N/I	N/I	
Hexachlorobenzene	70 ppb	0.1-2 ppb	4 ppb	N/R	2.5 ppb		<1-5 ppb	2-15 ppb	N/I	N/R	
Lindane	80 ppb	0.1-2 ppb	4 ppb	N/R	2.5 ppb		<1-5 ppb	2-15 ppb	N/I	2 ppb	
Mirex	2,000 ppb	N/I	N/I	N/R	2.5 ppb		<1-5 ppb	2-15 ppb	N/I	N/I	
Toxaphene	100 ppb	3-15 ppb	60 ppb	N/I	N/I		60 ppb	153 ppb	N/I	100 ppb	
Organophosphate Pesticides						N/I					1-15 ppb
Chlorpyrifos	30,000 ppb	N/I	N/I	N/R	2.5 ppb		N/I	N/I	N/I	10 ppb	
Diazinon	900 ppb	N/I	N/I	N/I	N/I		N/I	N/I	N/I	50 ppb	
Disulfoton	500 ppb	N/I	N/I	N/I	N/I		N/I	N/I	N/I	N/I	
Ethion	5,000 ppb	N/I	N/I	N/I	N/I		N/I	N/I	N/I	20 ppb	
Terbufos	10,000 ppb	N/I	N/I	N/I	N/I		N/I	N/I	N/I	N/I	
Chlorophenoxy Herbicides											
Oxyfluorfen	800 ppb	N/I	N/I	N/I	N/I	N/I	N/I	N/I	N/I	N/I	N/I
PAHs^x	10 ppb	20-100 ppb ^y	330 ppb ^y	N/I	N/I	N/I	N/I	N/I	N/I	N/R ^z	10 ppb
PCBs (total Aroclors)	10 ppb	(1-5 ppb) ^{aa}	(20 ppb) ^{aa}	N/R	(1.25-6.25 ppb) ^{bb}				50 ppb		
Aroclor 1016											20 ppb
Aroclor 1221											20 ppb
Aroclor 1232											20 ppb
Aroclor 1242		N/I	N/I	N/I	N/I		N/R	N/R	N/I	N/I	20 ppb
Aroclor 1248		N/I	N/I	N/I	N/I		62 ppb	167 ppb	N/I	50 ppb	20 ppb
Aroclor 1254		N/I	N/I	N/I	N/I		41 ppb	111 ppb	50 ppb	50 ppb	20 ppb
Aroclor 1260		N/I	N/I	N/I	N/I		61 ppb	155 ppb	50 ppb	50 ppb	20 ppb

See notes and references at end of table.

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Table J-1 (continued)

Target Analyte	SV ^b	Methods									
		Puget Sound Protocols ^c		National Study of Chemical Residues in Fish ^d		EMSL ^e	National Contaminant Biomonitoring Program ^f		California OEHHA ^g	State of California, Dept. of Fish and Game Environmental Services Division ^h	EPA 301(h) Monitoring Program ⁱ
		LOD ^j	PQL ^k	MLD ^l	TQL ^m	MDL ⁿ	LOD ^o	LOQ ^p	MDL ^q	LOD ^r	Detection Limits ^s
Dioxins/furans (total)^{cc} TCDD/TCDF PeCDD/PeCDF HxCDD/HxCDF HpCDD/HpCDF OCDD/OCDF	0.7 ppt	N/I	N/I	N/I 1 ppt 2 ppt 4 ppt 10 ppt N/I	N/R N/R N/R N/R N/I	N/I	N/I	N/I	N/I	N/I	N/I

CVAA = Cold vapor atomic absorption spectrophotometry.

GFAA = Graphite furnace atomic absorption spectrophotometry.

ICP = Inductively coupled plasma atomic emission spectrometry.

N/I = Target analyte not included in monitoring program or recommended methods.

N/R = Not reported.

PAHs = Polycyclic aromatic hydrocarbons.

PCBs = Polychlorinated biphenyls.

^a All values for SVs, detection limits, and quantitation limits are given in units of weight of analyte per wet weight of edible fish/shellfish tissue, unless otherwise noted.

^b From Table 5-2. Except for mercury, SVs are for general adult population using oral RfDs or SFs available in the EPA IRIS database and assuming a consumption rate (CR) = 6.5 g/d, average body weight (BW) = 70 kg, lifetime (70-yr) exposure, and for carcinogens a risk level (RL) = 10^{-5} . The RfD of 3×10^{-4} mg/kg/d for chronic systemic effects of methylmercury that was listed in IRIS through April 1995 was lowered by a factor of 5 to calculate the recommended SV of 0.6 ppm to account for a possible fivefold increase in fetal sensitivity to methylmercury exposure (WHO, 1990). This approach is consistent with, but somewhat more protective than, use of the current IRIS (1995) RfD of 1×10^{-5} mg/kg/d for the developmental effects of methylmercury (see Section 5.3.1.2).

Note Increasing CR, decreasing BW and/or using an RL $< 10^{-5}$ will decrease the SV. Program managers must ensure that detection and quantitation limits of analytical methods are sufficient to allow reliable quantitation of target analytes at or below selected SVs. If analytical methodology is not sensitive enough to reliably quantitate target analytes at or below selected SVs (e.g., PCBs, dioxins/furans), the program managers must determine appropriate fish consumption guidance based on lowest detectable concentrations, or provide justification for adjusting SVs to values at or above achievable method detection or quantitation limits.

^c Puget Sound Estuary Program (1990a,b). Analysis of total arsenic cadmium, and selenium by GFAA. Analysis of mercury by CVAA. Analysis of organochlorine and organophosphate pesticides and PCBs by gas chromatography/electron capture detection (GC/ECD). Analysis of PAHs by gas chromatography/mass spectrometry (GC/MS). Inorganic protocols based on U.S. EPA SW-846 methods (U.S. EPA, 1986b) and U.S. EPA Contract Laboratory Methods (U.S. EPA, 1987a). Organic protocols based on Krahn et al. (1988), U.S. EPA (1984, 1986b, 1988, 1989d), Horwitz et al. (1980), NUS (1985), MacLeod et al. (1985), and Brown et al. (1985), on a series of Puget Sound Estuary Program Workshops, and on a national QA Workshop sponsored by the National Oceanic and Atmospheric Administration (NOAA) and National Institute of Standards and Technology (NIST).

^d National Study of Chemical Residues in Fish (U.S. EPA, 1992a, 1992b). Analysis of mercury by CVAA (U.S. EPA, 1989a). Analysis of organochlorine and organophosphate pesticides and PCBs by GC/MS (U.S. EPA, 1989c). Analysis of dioxins/dibenzofurans by high-resolution gas chromatography/high-resolution mass spectrometry (HRGC/HRMS) (U.S. EPA, 1989b).

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- ^e U.S. EPA (1991). Analysis of total arsenic, cadmium, and selenium by ICP. Analysis of mercury by CVAA. MDLs determined in fish tissue matrix.
- ^f U.S. Fish and Wildlife Service National Contaminant Biomonitoring Program (Schmitt and Brumbaugh, 1990; Schmitt et al., 1990). Analysis of cadmium by GFAA. Analysis of mercury by CVAA. Analysis of total arsenic and selenium by hydride generation atomic absorption (HAA). Analysis of organochlorine pesticides and PCBs by GC/ECD.
- ^g Pollock et al. (1991). Composited fish samples extracted and analyzed for organics by GC/ECD using FDA Method PAM 211.1 in the *Pesticide Analytical Manual*--Vol. I (U.S. FDA, 1978). This method has been validated in interlaboratory studies and is an official method of the Association of Official Analytical Chemists (AOAC) for DDT, chlordane, and PCBs in fish. Mercury was determined using the AOAC flameless atomic absorption method (CVAA) (Williams, 1984). Analysis of tributyltin by GFAA.
- ^h California Department of Fish and Game. (1990). Metals methods based in part on EPA SW-846 methods (U.S. EPA, 1986b). Analysis of cadmium by flame AA and GFAA. Analysis of mercury by CVAA. Analysis of total arsenic and selenium by HAA. Analysis of tributyltin by GFAA. Organics methods based on FDA methods (U.S. FDA, 1975) and EPA 301(h) methods (U.S. EPA, 1986a). Analysis of organochlorine and organophosphate pesticides and PCBs by GC/ECD. Analysis of PAHs by gas chromatography/flame ionization detection (GC/FID).
- ⁱ U.S. EPA (1985, 1986a, 1987b). Analysis of total arsenic by GFAA, ICP, or HAA. Analysis of cadmium by GFAA or ICP. Analysis of selenium by GFAA or HAA. Analysis of mercury by CVAA. Analysis of organochlorine pesticides and PCBs by GC/ECD. Analysis of organophosphate pesticides by GC/phosphorus specific flame photometric or alkali flame ionization detection. Analysis of PAHs by GC/MS. Extract cleanup (e.g., removal of polar interferences by alumina column chromatography) assumed.
- ^j LOD = Limit of detection. Method detection limit as defined in 40 CFR 136 using a minimum of three replicates.
- ^k PQL = Practical quantitation limit. Defined in the Puget Sound Estuary Program as the minimum concentration of an analyte required to be measured and allowed to be reported without qualification as an estimated quantity for samples without substantial interferences. Based on the lowest concentration of the initial calibration curve (C, in µg/mL), the amount of sample typically analyzed (W, in g), and the final extract volume (V, in mL):
- $$\text{PQL } (\mu\text{g/g;ppm}) = \frac{C(\mu\text{g/mL}) \cdot V(\text{mL})}{W(\text{g})} .$$
- ^l MLD = Minimum level of detection. Concentration predicted from ratio of baseline noise area to labeled internal standard plus three times the standard error of the estimate from the weighted initial calibration curve.
- ^m TQL = Target quantitation limit. Specific detection limits were not determined for individual samples, so were operationally set at zero.
- ⁿ MDL = Method detection limit. Minimum concentration of an analyte that can be identified, measured, and reported with 99 percent confidence that the analyte concentration is greater than zero. Determined according to the procedure in 40 CFR 136 using seven replicates.
- ^o LOD (for metals) = $3\{S_b^2 - S_s^2\}$, where S_b^2 and S_s^2 are variances of concentrations measured for procedural blanks and a low-level sample, respectively. LOD (for pesticides) = Mean method blank plus three times the standard deviation. Determined according to Keith et al. (1983).
- ^p LOQ = Limit of quantitation. Mean method blank plus 10 times the standard deviation. Determined according to Keith et al. (1983).
- ^q MDL = Method detection limit. Determined according to procedure in 49 CFR 209, except for tributyltin, which was determined as 2 times the standard deviation of the sample blank.

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- ^r LOD = Limit of detection. The lowest concentration that is statistically different from a blank. Determined according to the IUPAC method in Long and Winefordner (1983).
- ^s From U.S. EPA (1985). Based on detection levels normally achieved in methods commonly used for tissue analyses in environmental laboratories. These detection limits are generally between the instrument detection limit (IDL) and method detection limit (MDL) (see Section 8.3.3.3) and are based on the expertise and best professional judgment of experienced analysts. Detection limits for metals based on 5 g (wet weight) of muscle tissue digested and diluted to 50 mL. Detection limits for organics based on 25 g (wet weight) of muscle tissue extracted, concentrated to 0.5 mL after gel permeation chromatography cleanup, and 1 µL injected. Bonded, fused silica capillary GC columns, which provide better resolution than packed columns, are assumed for analysis of semivolatile compounds.
- ^t Inorganic arsenic was not included in any of the major monitoring programs (see Appendix C). Detection limits for the analysis of inorganic arsenic by hydride generation atomic absorption (HAA) and high-performance liquid chromatography-inductively coupled plasma mass spectrometry (HPLC-GC/MS) are on the order of 5 -50 ppb and 50-100 ppb, respectively (E. Crecelius, Battelle Pacific Northwest Laboratories, Marine Sciences Laboratory, Sequim, WA, personal communication, June 1995).
- ^u LOD = Limit of detection. No procedure given for determining the LOD.
- ^v GFAA method used is not specific for tributyltin. Mono-, di-, and tetrabutyltin as well as other alkyltins may be included in the analysis. Method detection limit for analysis by gas chromatography/flame photometric detection (GC/FPD), which is specific for tributyltin, is 2-5 ppb (E. Crecelius, Battelle Pacific Northwest Laboratories, Marine Sciences Laboratory, Sequim, WA, personal communication, June 1995).
- ^w The higher detection limits are appropriate for pesticides such as mirex, the DDTs, and endosulfans. Compounds such as lindane and hexachlorobenzene can be detected at the lower limits. Toxaphene (a mixture) may require a higher detection limit than the other organochlorine pesticides.
- ^x All tabulated detection limits for PAHs are based on low-resolution GC/MS. Detection limits of less than 1 ppb can be achieved using HRGC/HRMS.
- ^y Values given are for semivolatile organics in general.
- ^z Dry weight detection limits for 24 PAHs in fish tissue ranged from 0.1 to 0.2 ppb.
- ^{aa} Aroclors not determined. Values given are for individual mono- through decachlorobiphenyls.
- ^{bb} Aroclors not determined. PCBs reported by total congener at the following levels of chlorination (TQLS in parentheses): 1-3 (1.25 ppb); 4-6 (2.5 ppb); 7-8 (3.75 ppb); 9-10 (6.25 ppb).
- ^{cc} Detection and quantitation limits obtained from a survey of 10 laboratories with expertise in dioxin/dibenzofuran analyses by HRGC/HRMS ranged from 0.04-10 ppt and 0.2-100 ppt, respectively.

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